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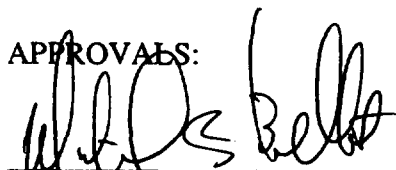
**ADDENDUM NO. 5  
QUALITY ASSURANCE PROJECT PLAN  
QUARTERLY GROUNDWATER MONITORING**

**FOR THE**

**BLACKWELL LANDFILL  
DUPAGE COUNTY, ILLINOIS**

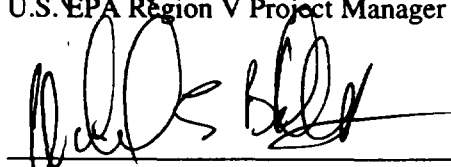
**April 1998**

APPROVALS:



U.S. EPA Region V Project Manager

Montgomery Watson QA Officer



U.S. EPA Region V Quality Assurance Reviewer

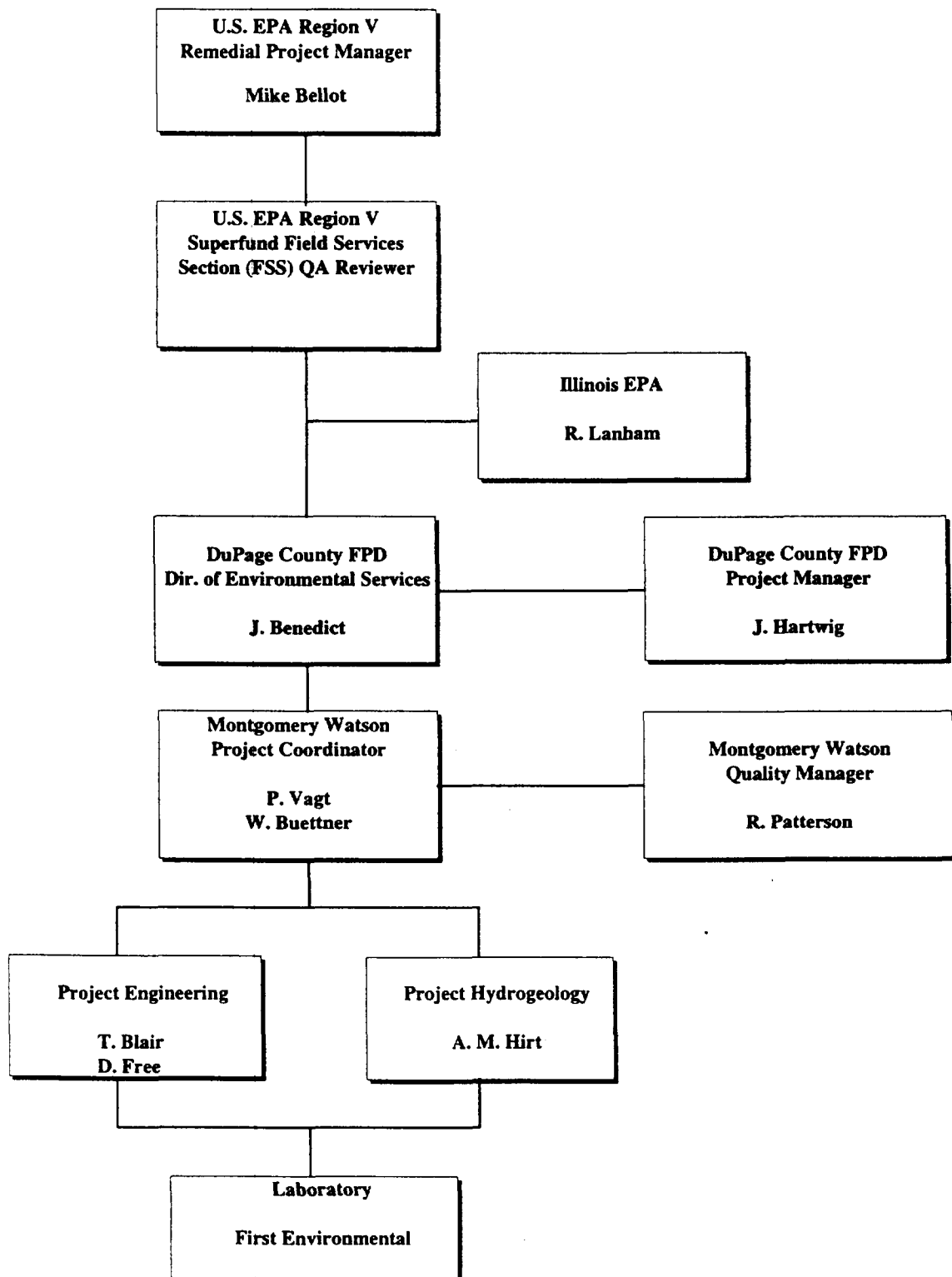
First Environmental Laboratory QA Officer

Forest Preserve District Project Manager

First Environmental Laboratory Director

Montgomery Watson Project Manager

**FIGURE 1**  
**ORGANIZATION CHART**  
**BLACKWELL LANDFILL RESPONSE ACTION**



## First Environmental Laboratories

### Standard Operating Procedure

**Title:** Semi-Volatile Organics Analysis; Method 8270C  
Polynuclear Aromatic Hydrocarbons (PNAs or PAHs)

**Regulatory References:** SW-846; 8270C

**Regulatory Limits:** Varies

**Preservation Requirements:** Cool, 4°C

**Container:** One quart amber or 16 oz wide mouth with Teflon lined closure.

**Single Analysis Sample Volume:** 1000mL or ~30g.

**Holding Time:** 7 Days from sample collection (aqueous).  
14 Days from sample collection (non-aqueous).  
35 Days from sample extraction.

**(Range) Reporting Limit:** 0.13 to 10 ug/L (ground water samples)  
8 to 50 ug/kg (soil/sediment samples).

Sample reporting limits are highly matrix dependent and will be proportionally higher for sample extracts that require dilution to avoid saturation of the detector.

Routine Reporting Limits		
	Aqueous ug/L	Non-Aqueous ug/kg
Acenaphthene	10	50
Acenaphthylene	10	50
Anthracene	5	50
Benzo[a]anthracene	0.13	8.7
Benzo[b]fluoranthene	0.18	11
Benzo[k]fluoranthene	0.17	11
Benzo[g,h,i]perylene	0.4	50
Benzo[a]pyrene	0.2	15
Chrysene	1.5	50
Dibenz[a,h]anthracene	0.3	20
Fluoranthene	2	50
Fluorene	2	50
Indeno[1,2,3-cd]pyrene	0.3	29
Naphthalene	10	25
Phenanthrene	5	50
Pyrene	2	50

Table 1

## Summary of Method:

Method 8270C is used to quantitate most neutral compounds (such as PNAs) that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. The GC is programmed to separate the components, which are detected with a mass spectrometer. Prior to using this method, the samples should be prepared by using the appropriate sample preparation and clean-up methods.

## 1. Instrumentation / Apparatus / Glassware

For a comprehensive listing of the instrumentation, apparatus and glassware required for this method see "Test Methods for Evaluating Solid Waste", SW-846, Method 8270C Section 4.0.

The analyst should be familiar with the operation of the Hewlett Packard Enviroquant data acquisition and reduction software before beginning the analysis of samples. The analyst should also be familiar with the analytical instrumentation and have direct access to any operational manuals. The analyst should have read and be familiar with the entire contents of method 8270C. A copy of the method should be available for reference.

## Instrument Conditions:

### GC/MS Instrument Conditions (Splitless Injection)

In this technique the electronically controlled injection port pressure is programmed to allow the maximum amount of analyte to be introduced to the analytical column. The sample is introduced to the narrow bore column using the splitless injection technique.

Parameter	Setting
Column	HP-5MS 30meter
Column ID/Thickness	0.25mm ID 1.12um
Interface	Cap Direct Splitless Inj.
GC Type	HP5890 Series II/Plus
Mass Spectrometer	HP5972A
Tune	DFTPP Tune
Scan Range	35-510 amu
Sampling #	3
Threshold	100
Carrier Gas	Helium
Vacuum Comp.	On
Pressure(1)	20psi @ 50 deg C
Time Pressure(1)	1.0 min
Pressure Rate(1)	99 psi/min
Pressure(2)	5.8 psi
Constant Flow Mode	0.84 mL/min
Inj. B Temp	220 C
Detector B Temp	280 C
Initial Temp	50 C

Parameter	Setting
Initial Time	3 minutes
Rate(1)	10.00 deg C/min
Final Temp(1)	200 deg C
Final Time(1)	0 min
Rate(2)	20.0 deg C/min
Final Temp(2)	290 deg C
Final Time(2)	10.0 min
Total GC run time	32.50 min
Purge B	Off (for 1.0 min)

Table 2

## 2. Standards

### DFTPP System Check Standard

The DFTPP system check standard is purchased prepared from Supelco (Catalog #4-7387). This solution contains DFTPP, 4,4'-DDT, Pentachlorophenol and Benzidine at 50 ug/mL of each component.

### Surrogate Spike:

Prepare the surrogate spiking solutions for Base/Neutral (PNA) fractions.

Dilute 5.0mL of the Base/Neutral Surrogate Standard Mix (Restek catalog #31086) to 250 mL with methanol and place into a clean 8 ounce amber jar with a teflon closure. This solution should be stored at 4°C. PNA samples should be spiked with 100 uL of this solution. The expected sample concentration of the surrogate compounds is 10 ug/L.

### MS/MSD Spike:

Prepare a solution of the B/N (PNA) spiking compounds. Dilute 1.0mL of the B/N Matrix Spike Mix (Restek #31074) to 100 mLs in methanol. PNA samples should be spiked with 100 uL of this solution. The expected sample concentration of each spiking compound is 2.0 ug/L.

### Internal Standard Solution

The internal standard solution for BNA extracts is prepared first by sonicating the ISTD mix (RK#31006, which contains 4000 ug/mL of each ISTD compound) for 10 minutes. If particulates still appear in the bottom of the ISTD ampule, sonicate the solution longer until these particles go into solution. Then dilute 1.0 mL of the sonicated ISTD mix to a 10.0 mL final volume. This is your spiking solution. Prior to spiking samples or standards, sonicate this solution for 10 minutes. Then spike each 1.0 mL of PNA extract with 25.0 uLs of this solution. This will result in an extract concentration of 10 ug/mL for each individual ISTD component.

The internal standard solution includes the following compounds; 1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, and Perylene-d12.

## PNA Initial Calibration Preparation

Prepare the following mother solution:

### PNA Mother Solution

Calibration Mixture	Catalog #	Initial Conc	Volume	Final Conc
SV Calibration Mix #5 (PNAs)	RK31011	2000 ug/mL	1.0mL	400 ug/mL
B/N Surrogate Standard Mix	RK31024	1000	1.0	200 ug/mL
Methylene Chloride (solvent)			3.0	

Table 3

Prepare 10mLs of a 20.0 ug/mL Standard. This is the high point of the 5-point calibration curve.

- Dilute 500uLs of the PNA mother solution to 10.0 mLs with methylene chloride.

The remaining four points of the calibration curve are made by doing a serial dilution on the 20.0 ug/mL standard. Prepare the serial dilutions according to the following table:

Calibration Solution	0.25 ug/mL	0.50 ug/mL	1.0 ug/mL	5.0 ug/mL
5.0 ug/mL PNA Std	12.5 uLs	25 uLs	50 uLs	250 uLs
uLs Methylene Chloride	987.5	975	950	750

Table 4

Prior to analysis add the appropriate amount of PNA ISTD solution to each vial. (25uL of a 400ug/mL solution will yield 10 ug/mL).

Analyze the initial calibration curve according to the analysis procedure in Section 4.

Tabulate the table of response factors (RF) for each compound. The RF is calculated as follows:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

where:

$A_x$	=	Area of characteristic ion for the compound being measured.
$A_{is}$	=	Area of the characteristic ion for the specific internal standard.
$C_{is}$	=	Concentration of the specific internal standard.
$C_x$	=	Concentration of the compound being measured.

The RF, the average RF and the %RSD are automatically calculated with the data system software. The initial calibration response factor report should be printed and included with the daily QC file folder. The 5 SPCC compounds should be evaluated and meet the minimum response factors listed in Table 6. The %RSD for each of the CCC compounds listed in Table 5 must be less than 30% in the initial calibration.

Compound	Minimum RF Criteria	Initial Calib Max %RSD	Cont. Calib Max %RSD
Acenaphthene	0.050	30	20
Fluoranthene	0.050	30	20
Benzo(a)pyrene	0.050	30	20

Table 5

### 3. Safety

See appropriate section in Safety SOP.

### 4. Analytical Procedures

4.1 **Tune the GC/MS system** using "Target Tune". Store the tune values in the file named "dftpp.u". Store the hardcopy output in the daily QA/QC file folder.

4.2. **Analyze Tuning Compound DFTPP:** Prior to the analysis of any standards or samples, a DFTPP must be analyzed and meet quality criteria. This must be done at the start of every 12-hour batch. Inject 1.0 uL of DFTPP tuning solution (25 ng/uL) into the gas chromatograph injection port. When setting up your batch sequence, indicate that this run is "DFTPP" in the sample type field. This will automatically evaluate the DFTPP run and issue a report. In this report, all ions should indicate "PASS". If not, re-tune as in 4.1 above and repeat the DFTPP analysis. The mass spectrum of the DFTPP must meet the following criteria:

Mass	Intensity Required (relative abundance)
51	30 to 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 to 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 to 9% of mass 198
275	10 to 30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17 to 23% of mass 442

Table 6

4.3 **Analyze Initial Calibration Standards.** Analyze the 5-point calibration mixtures that are detailed in Section 2; Table 3 or 4. Analyze these samples using the conditions and method parameters detailed in Section 1.

4.4 The percent relative deviation (%RSD) should be less than 15% for all compounds. The %RSD for each CCC compound (Table 4 from method 8270C) must be less than 30%. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units.

**4.5 Analyze Daily Continuing Calibration Standard:** Prepare and analyze the 50.0 ppm concentration standard. Analyze this sample using the conditions and method parameters detailed in section 1.

To evaluate the results of this run enter the ENVDA program. Select "File" and load the correct data file. Select "Method" and load the proper method, 8270a.m. Select the option "Concal", then select "Evaluate Data as Continuing Calibration to Printer". This will generate a report that compares this standard analysis to the initial calibration curve. All of the continuing calibration compounds should all be less than 20% (Compounds above 20% will be indicated with a "#" on this report). If any of these are out of control, re-run the standard or prepare a new 5-point calibration. (Check the areas of the peaks with QEDIT to be sure they have been integrated properly) A Continuing Calibration Standard must be analyzed every 12 hours during analysis.

**4.6 Analyze Extraction Method Blank:** Analyze this sample using the conditions and method parameters detailed in section 1.

Evaluate the Method Blank. No target compounds should be present. Proceed with sample analysis if all indicators are within control limits.

**4.7 Analyze each sample.** Analyze each standard, blank, MS, MSD, LCS and sample using the conditions and method parameters detailed in section 1. Evaluate each sample chromatogram for the presence/absence of target compound analytes.

**4.7.1 Sample Calculations.** Calculate the concentration of each identified analyte in the sample as follows:

$$\text{Water Concentration (ug/L)} = (A_x) (I_s) (V_{ex}) / (A_{is}) (RF) (V_o)$$

where:

$A_x$	=	Area of characteristic ion for compound being measured
$I_s$	=	Amount of internal standard being injected (ng).
$A_{is}$	=	Area of characteristic ion for the internal standard
RF	=	Mean relative response factor for compound being measured.
$V_o$	=	Volume of liquid extracted taking into consideration any dilutions made.
$V_{ex}$	=	Volume of final extract (mL)

$$\text{Soil Concentration (ug/kg)} = (A_x) (I_s) (V_e) / (A_{is}) (RF) (W_s) (D)$$

where:

$A_x$	=	Area of characteristic ion for compound being measured
$I_s$	=	Amount of internal standard being injected (ng).
$A_{is}$	=	Area of characteristic ion for the internal standard
RF	=	Mean relative response factor for compound being measured.
$V_e$	=	Volume of final extract (uL)
$W_s$	=	Weight of sample extracted (g).
D	=	% dry weight of sample/100, or 1 if sample is to be reported on a wet-weight basis.

**4.8 Analyze a MS/MSD pair for every 20 sample analyses or according to the frequency dictated by the QAPP for the sample project..** The MS/MSD pair should be evaluated for accuracy (% recovery) and precision (%RPD). The results should be tabulated and included in the daily QC package. The following sub-set compounds should be used. Recovery criteria will be established and updated by

determining the standard deviation every 30 samples. The UCL and LCL will be +/- 3 times the standard deviation. Control charts with this data will be maintained and updated on a routine basis. The control limits must fall within the range detailed on the following table:

Spike Compound	% Rec Limits	% RPD
Acenaphthene	30-125	25
Acenaphthylene	46-118	31
Anthracene	30-125	25
Benzo[a]anthracene	30-125	25
Benzo[b]fluoranthene	30-125	25
Benzo[k]fluoranthene	30-125	25
Benzo[g,h,i]perylene	30-125	25
Benzo[a]pyrene	30-125	25
Chrysene	30-125	25
Dibenz[a,h]anthracene	30-125	25
Fluoranthene	30-125	25
Fluorene	30-125	25
Indeno[1,2,3-cd]pyrene	30-125	25
Naphthalene	30-125	25
Phenanthrene	30-125	25
Pyrene	26-127	31

Table 7

## 5. Interferences

The analytical system should be checked to ensure freedom from interferences, under the analysis conditions, by analyzing method blanks. Cross-contamination can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by the analysis of solvent to check for carry-over contamination. The low-concentration sample that may have followed a high-concentration sample should be re-analyzed to confirm the analytical result. To reduce carry-over, the sample syringe should be rinsed out between sample injections.

## 6. Quality Control Indicator Assessment

DFTPP and a continuing calibration standard must be run every 12 hours prior to the analysis of samples.

A method blank should be analyzed with every analytical batch. No target compounds should be present

An MS/MSD pair should be extracted for every twenty samples.

A Laboratory Control Spike (LCS) may be required if the MS/MSD is outside control limits. An LCS should be extracted for every 20 samples in the event it will need to be evaluated. An LCS is an aliquot of laboratory DI water spiked with the MS/MSD spiking solution.

The internal standard responses and retention times in the analytical batch must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made as required. If the EICP area for any one of the internal standards changes by a factor of two (-50% to +100%) from the last daily calibration check standard, the

mass spectrometer must be inspected for malfunctions and corrections must be made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

Surrogate compounds will be added to every standard, blank and sample that is analyzed. The surrogate recoveries must fall within the following specifications. If any one recovery is outside these limits, the sample must be re-analyzed to establish that a sample matrix effect is present.

Surrogate Compound	Low/High Water	Low/High Soil/Sediment
Nitrobenzene-d5	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
Terphenyl-d14	33-141	18-137

Table 8

## 7. Notes

**Routine Maintenance.** The analyst should be familiar with the routine maintenance procedures for both the chromatographic and mass spectrometer systems. Routine maintenance should include, but not be limited to, septum changes, column cutting, mass spectrometer source cleaning, vacuum pump fluid replacement and GC inlet cleaning. The analyst must be aware of system indicators and quality control indicators that warrant attention to these maintenance procedures. The analyst should contact a supervisor if there is any question about these procedures.

For the analysis of PNAs by method 8270, we analyze and evaluate the data for only the target compounds of interest. The initial calibration mixture, continuing calibration mixture and MS/MSD solutions contain only a sub-set of the entire 8270 list. The QA/QC performed on this analysis is used to evaluate only the 16 PNA compounds.

The %RPD and Response Factors for the PNA compounds are evaluated in the initial and continuing calibration standards. These are evaluated to insure that there is no degradation in the standard material or in the chromatographic system. Since the analysis is only interested in the PNA compounds, it is only these compounds that are evaluated.

## 8. Approvals

Reviewed for Technical Accuracy by: 

Reviewed for Quality Assurance Compliance by: 

Implementation Date: 4/15/98

End Use Date: \_\_\_\_\_



16 April 1998

Mr. William Mottashed  
First Environmental Laboratories, Inc.  
1600 Shore Road  
Naperville, IL 60563

1265 Capital Airport Drive  
Springfield, IL 62707-8413

Phone: 217-753-1148  
FAX: 217-753-1152

RE: SOP Data request

Dear Mr. Mottashed:

In response to your request regarding the modification to our Standard Operating Procedure for Method 525.2, the following comments have been prepared to clarify the highlighted comments on your document.


1. Comment: GC/MS data system characteristic should be addressed in the SOP.  
Response: A copy of the QAQC referenced in the SOP is attached hereto (see attachment #1).
2. Comment: Sample collection should be part of the SOP.  
Response: A copy of Section 5 of the QAQC provides the sampling protocol required for Method 525.2 (see attachment #2). Its absence from the approved SOP for 525.2 is because we do not provide sampling services for our clients.
3. Comment: Referenced Method requires sample extraction within 7 days.  
Response: The statement highlighted in your document is incorrect. The Method clearly states stability for a 14 day period especially for the compounds that are applicable to this project. The method also indicates that there are special circumstances for the following compounds, none of which are included in this project:

Merphos  
Disulfoton  
Diazinon  
Turbufos

Carboxin  
Disulfoton sulfoxide  
Fenamiphos

If you have any questions, please feel free to contact our office.

Sincerely,

  
Stephen R. Johnson  
Laboratory Director

### 3.2 Laboratory Equipment

The primary analytical instrumentation consists of the following instruments or equipment listed by the area within the facility:

#### Volatile Organic Area

Hewlett-Packard 5890 Series II Gas Chromatograph with Tandem Ol Analytical 5240 Photoionization Detector/Electrolytic Conductivity Detector (PID/ELCD); Capillary Injectors; TekMar 3000 Purge and Trap with Hand Held Controller; TekMar 2016-16 position Autosampler; Hewlett-Packard 3396B Integrator - Dual Channel.

Hewlett-Packard 5972 MSD Volatile System w HPIB includes: 5972 Detector; G1034C MS Software; IBM Compatible 486/66mhz Computer; 8MB RAM; 430 MB Hard Drive; 3.5" Floppy Drive; 120MB Tape Back-up; Sony VGA Color Monitor; keyboard and mouse; HPIB; Laser Jet 4 Printer; HP Ion Gauge Controller; HP Enviroquant Software; HP NIST Library; HP 5890 Series II Gas Chromatograph; Packed Injector; Jet Separator Makeup Gas Kit; Tekmar 3000 Purge and Trap Purge and Trap Concentrator; and a Tekmar Precept II Autosampler with heated purge pockets.

#### Semi-Volatile Organic Area

Hewlett-Packard 5890 Series II Gas Chromatograph with Electron Capture Detector (ECD) and Nitrogen Phosphorus Detector (NPD); split/splitless injectors; INET Communication; Pressure Regulators; HP 3396B integrator - Dual Channel.

Hewlett-Packard 5972 MSD/5890 Series II Gas Chromatograph Semi-Volatile System w HPIB includes: 5972 Detector; G1034C MS Software; IBM Compatible 486/66mhz Computer; 8MB RAM; 430 MB Hard Drive; 3.5" Floppy Drive; 120MB Tape Back-up; Sony VGA Color Monitor; keyboard and mouse; HPIB; Laser Jet 4 Printer; HP Ion Gauge Controller; HP Enviroquant Software; HP NIST Library; HP 5890 Series II Gas Chromatograph; Split/splitless Injector with EPC, EPC Board. HPIB Communication, HP 7673 Autosampler - Single Tower.

Hewlett-Packard 5973 MSD/6890 Semi-Volatile System w/ECD includes: 5793 Mass Selector Detector, an Electron Capture Detector (ECD) w/EPC; G1036A NIST Chemical Library; 1038A Pesticide MS Spectral Library; G1020D MS ChemStation; HP Vectra XM5/150mhz computer; 32 MB

Ram; 2.3 GB Hard Drive; 3.5" Floppy and CD ROM Drive; Mouse & Keyboard; HP Ultra VGA 1280 Monitor; H-P Laserjet 5 Printer; ion gauge controller; HP 6890 Series Injector/Autosampler; Split/Splitless Injector with EPC

Hewlett-Packard 1050 HPLC System includes: HPLC Chemstation Software on H-P 586/100mhz, 16MB RAM, 540 MB Hard Drive Computer, 17" VGA Monitor, keyboard & Mouse; w/ Quaternary pump, programmable sampler, diode-array detector, programmable fluorescence detector, and PAH Column Sum.

#### Wet Chemistry & Inorganic Area

Hewlett Packard 4500 ICP/MS, w/HP Vectra Pentium PC and ICP/MS Software; sample probe wash pump; CETAC auto-autosampler; air-cooled non-CPC water chiller.

Perkin-Elmer 4100ZL Atomic Absorption Spectrometer with Fume Extractor and Cooling System and 4100ZL System Controller Assembly, 2 lamp EDL Power supply.

Perkin-Elmer Plasma 400 ICP, P-400, Controller, P-400 Software. Okidata 320 Printer, Perkin-Elmer AS-90 Autosampler.

Dionex DC-120 Ion Chromtography w/Dual Column and 4400 Integrator

Coleman Mercury Analyzer, Model 50B

Orion Model 970A with 900A Printer

Orion Model 290A pH/ISE meter

Orion Model 124 Conductivity/TDS Meter

Sartorius Model BP211D 5-place balance with computer/printer read-out

Sartorius Model BA61 Toploading Balance

Sartorius Model B1417-93 Analytical Balance

Sartorius Model LC420 Analytical Balance

Sequoia-Turner Model 340 Digital Spectrophotometer

Sonics and Materials Model VC375 Ultrasonic Processor

**5.9 Sample Collection, Preservation, and Storage - Method 525.2**

**5.9a Sample collection.** When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 2 - 5 minutes). Adjust the flow to about 500 ml/min and collect the samples from the flowing stream. Keep samples sealed from collection time until analysis. When sampling from an open body of water, fill the container with water from a representative area. Sampling equipment, including automatic samplers, must be free from plastic tubing, gaskets, and other parts that may leach analytes into the water. Automatic samplers that composite samples over time must use refrigerated glass sample containers.

**5.9b Sample dechlorination and preservation.** All samples should be iced or refrigerated at 4°C from the time of collection until extraction. Residual chlorine should be reduced at the sampling site by addition of reducing agent. Add 40-50 mg of sodium sulfite or sodium arsenite (these may be added as solids with stirring until dissolved) to each liter of water. Hydrochloric acid should be used at the sampling site to retard the microbiological degradation of some analytes in unchlorinated water. The sample pH is adjusted to <2 with 6 N hydrochloric acid. This is the same pH used in the extraction, and is required to support the recovery of pentachlorophenol.

**5.9c Holding time.** Samples must be extracted within 14 days and extracts analyzed within 30 days of sample collection.

**5.9d Field blanks**

1. Processing of a field reagent blank (FRB) is recommended along with each sample set, which is composed of the samples collected from the same general sample site at approximately the same time. At the laboratory, fill a sample container with reagent water, seal, and ship to the sample site along with the empty sample containers. Return the FRB to the laboratory with the filled sample bottles.

2. When hydrochloric acid is added to samples, use the same procedures to add the same amount to the FRB.

**METHOD 525.2 DETERMINATION OF ORGANIC COMPOUNDS IN DRINKING WATER  
BY LIQUID-SOLID EXTRACTION AND CAPILLARY COLUMN  
GAS CHROMATOGRAPHY/MASS SPECTROMETRY**

Revision 1.0

March 1994

J.W. Eichelberger, T.D. Behymer, W.L. Budde - Method 525,  
Revision 1.0, 2.0, 2.1 (1988)

J.W. Eichelberger, T.D. Behymer, and W.L. Budde - Method 525.1  
Revision 2.2 (July 1991)

J.W. Eichelberger, J.W. Munch, and J.A. Shoemaker  
Method 525.2 Revision 1.0 (February, 1994)

**ENVIRONMENTAL MONITORING SYSTEMS LABORATORY  
OFFICE OF RESEARCH AND DEVELOPMENT  
U.S. ENVIRONMENTAL PROTECTION AGENCY  
CINCINNATI, OHIO 45268**

determined, the samples cannot be held for 14 days but must be extracted immediately after collection and preservation: carboxin, diazinon, disulfoton, disulfoton sulfoxide, fenamiphos, and terbufos.

#### 8.4 Field blanks.

8.4.1 Processing of a field reagent blank (FRB) is recommended along with each sample set, which is composed of the samples collected from the same general sample site at approximately the same time. At the laboratory, fill a sample container with reagent water, seal, and ship to the sampling site along with the empty sample containers. Return the FRB to the laboratory with the filled sample bottles.

8.4.2 When sodium sulfite and hydrochloric acid are added to samples, use the same procedure to add the same amounts to the FRB.

### 9. QUALITY CONTROL

9.1 Quality control (QC) requirements are the initial demonstration of laboratory capability followed by regular analyses of laboratory reagent blanks, laboratory fortified blanks, and laboratory fortified matrix samples. The laboratory must maintain records to document the quality of the data generated. Additional quality control practices are recommended.

9.2 Initial demonstration of low disk or cartridge system background. Before any samples are analyzed, or any time a new supply of cartridges or disks is received from a supplier, it must be demonstrated that a laboratory reagent blank (LRB) is reasonably free of contamination that would prevent the determination of any analyte of concern. In this same experiment, it must be demonstrated that the particle size and packing of the LSE cartridges or the preparation of the disks are acceptable. Consistent flow rate with all samples is an indication of acceptable particle size distribution, packing, and proper preparation.

9.2.1 A source of potential contamination is the liquid-solid extraction (LSE) cartridge or disk which could contain phthalate esters, silicon compounds, and other contaminants that could prevent the determination of method analytes (5). Although disks are generally made of an inert matrix, they may still contain phthalate material. Generally, phthalate esters can be leached from the cartridges into ethyl acetate and methylene chloride and produce a variable background in the water sample. If the background contamination is sufficient to prevent accurate and precise measurements, the condition must be corrected before proceeding with the initial demonstration.

ml/min and collect samples from the flowing stream. Keep samples sealed from collection time until analysis. When sampling from an open body of water, fill the sample container with water from a representative area. Sampling equipment, including automatic samplers, must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample. Automatic samplers that composite samples over time should use refrigerated glass sample containers if possible.

- 8.2 Sample dechlorination and preservation. All samples should be iced or refrigerated at 4°C and kept in the dark from the time of collection until extraction. Residual chlorine should be reduced at the sampling site by addition of 40-50 mg of sodium sulfite (this may be added as a solid with stirring or shaking until dissolved) to each water sample. It is very important that the sample be dechlorinated prior to adding acid to lower the pH of the sample. Adding sodium sulfite and HCl to the sample bottles prior to shipping to the sampling site is not permitted. Hydrochloric acid should be used at the sampling site to retard the microbiological degradation of some analytes in water. The sample pH is adjusted to <2 with 6 N hydrochloric acid. This is the same pH used in the extraction, and is required to support the recovery of acidic compounds like pentachlorophenol.

8.2.1 If cyanazine is to be determined, a separate sample must be collected. Cyanazine degrades in the sample when it is stored under acidic conditions or when sodium sulfite is present in the stored sample. Samples collected for cyanazine determination MUST NOT be dechlorinated or acidified when collected. They should be iced or refrigerated as described above and analyzed within 14 days. However, these samples MUST be dechlorinated and acidified immediately prior to fortification with internal standards and extraction using the same quantities of acid and sodium sulfite described above.

8.2.2 Atraton and prometon are not efficiently extracted from water at pH 2 due to what appears to be their ionization in solution under acidic conditions. In order to determine these analytes accurately, a separate sample must be collected and dechlorinated with sodium sulfite, but no acid should be added. At neutral pH, these two compounds are recovered from water with efficiencies greater than 90%. The data in Tables 3, 4, 5, 6, and 8 are from samples extracted at pH 2.

- 8.3 Holding time. Results of the time/storage study of all method analytes showed that all but six compounds are stable for 14 days in water samples when the samples are dechlorinated, preserved, and stored as described in Sect. 8.2. Therefore, samples must be extracted within 14 days and the extracts analyzed within 30 days of sample collection. If the following analytes are to be